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LABORATORY CONFIRMATION OF METHODS OF DETERMINATION OF RESPIRABLE COMBUSTIBLE DUST

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Canadian Explosive Atmospheres Laboratory

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LABORATORY CONFIRMATION OF METHODS OF DETERMINATION OF RESPIRABLE COMBUSTIBLE DUST

by

E.D. Dainty*, J.A. Ebersole**, J. Vallieres*** and M.K. Gangal+

ABSTRACT

Because of significant differences in underground gravimetric measurements of respirable combustible dust (RCD), with and without the use of a pre-separating cyclone to pass only the respirable fraction, it was decided to make confirmatory laboratory determinations.

Three tests on each of three different days were conducted in a simulated mine environment produced by diluting the exhaust flow of a steam-cleaned Deutz F6L 912W engine by fresh air from the test cell ventilation system. It was determined that the average particulate material collected (derived from three cassettes per test), was 1.68 (-0.07/+0.11) mg when no pre-separating cyclone was used, and 1.73 (-0.07/+0.08) mg when a cyclone was placed upstream of each cassette in the sampling flow. These weights correspond to concentrations of 2.39 and 2.47 mg/m^3 respectively.

This represents an average mass difference of 0.05 mg, or 3%, which is less than the in-sample variation. This appears to be an excellent confirmation of the oft-used assumption, based on particle size

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KEYWORDS: diesel emissions, particulate, soot, cyclone.

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ABSTRACT (cont'd)

studies, that diesel particulates are 100% respirable. Therefore, the use of a cyclone should not produce a difference when only diesel soot is sampled.

The difference encountered during measurements in the mine must, therefore, be the result of the presence of combustibles other than soot in the environment. Such combustibles could be a component of the ore dust or water of hydration of the ore, or aerosols of drill oil mist, lubeoil, hydraulic oil, or fuel evaporated from hot machine surfaces. There is also a complicated possibility of soot agglomerating or depositing onto other larger-than-respirable airborne non-combustible particulates.

In addition, the performance of the prototype "Tapered Element Oscillating Microbalance (TEOM)" on loan from the United States Bureau of Mines, was evaluated. These preliminary checks indicated lower TEOM values of particulate concentration than the gravimetric method. At the low sampling flow of 0.12 litres/min with pre-separators in place, the gravimetric method gave a concentration of 2.66 mg/m^3 compared to 1.93 mg/m^3 for the TEOM. At a higher TEOM sampling flow of 2.0 litres/min, the results were 2.37 mg/m^3 compared to 1.26 mg/m^3 respectively. Thus, the instrument appears to be flow sensitive. The TEOM has a high mass resolution which was checked by weighing. The principle appears to be sound however, and the production model may prove to be the best available means for real-time soot measurements.

CONFIRMATION EN LABORATOIRE DE LA PRÉCISION DES MÉTHODES UTILISÉES POUR DÉTERMINER LA QUANTITÉ DE POUSSIÈRE INHALABLE PROVENANT DES COMBUSTIBLES

par

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RÉSUMÉ

Des différences importantes existent entre les mesures gravimétriques de la poussière inhalable provenant des combustibles (RCD), effectuées avec ou sans séparation préalable afin de ne laisser passer que la fraction inhalable. Pour ces raisons, nous avons décidé de faire des essais de laboratoire afin de confirmer la précision des méthodes utilisées.

Nous avons mené trois essais par jour pendant trois jours dans un environnement minier simulé, produit en diluant les gaz d'échappement d'un moteur Deutz F6L 912W nettoyé à la vapeur avec de l'air pur prevenant du système de ventilation du capteur d'échantillons. La quantité moyenne de matière recueillie (enregistrée sur trois cassettes pendant chacun des essais) était de 1,68 (-0,07/+0,11) mg sans séparation préalable, et de 1,73 (-0,07/+0,08) mg quand un séparateur était placé en amont de chaque cassette dans le débit d'échantillonnage. Les poids relevés correspondent à des concentrations de 2,39 et 2,47 mg/m^3, respectivement.

Ces mesures comportent une différence moyenne de 0,05 mg, ou 3 % (en poids), ce qui est inférieur à l'écart relevé au cours de l'échantillonnage. Ceci semble confirmer sans aucun doute l'hypothèse très connue découlant

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Mots-clé : gaz d'échappement de diesels, particule, suie, cyclone.

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d'études granulométriques, selon laquelle les particules provenant des diesels sont inhalables à 100 %. Par conséquent, l'utilisation d'un cyclone ne devrait marquer aucune différence quand l'échantillonnage ne porte que sur de la suie de diesel.

La différence remarquée au cours du mesurage dans la mine doit, par conséquent, être le résultat de la présence dans l'environnement de combustibles autres que la suie. De tels combustibles pourraient être des constituants de poussière de minerai ou de l'eau provenant de l'hydratation du minerai ou d'aérosols contenus dans les buées d'huile de forage, d'huile lubrifiante, d'huile hydraulique ou de combustible s'évaporant des surfaces chaudes des machines. Il existe également une possibiité que la suie puisse s'agglomérer ou se déposer sur d'autres particules non combustibles, plus grandes que les particules inhalables aéroportées.

De plus, nous avons évalué le rendement du prototype de la microbalance oscillante à élément conique (TEOM) prêtée par le United States Bureau of Mines. Les vérifications préliminaires indiquent que les valeurs relatives à la concentration de poussière relevées par la microbalance oscillante à élément conique sont moins élevées que celles établies au moyen de la méthode gravimétrique. Quand le débit de l'échantillonnage était peu élevé, soit 0,12 litres/min, et que les pré-séparateurs étaient en service, la méthode gravimétrique a permi de relever une concentration de 2,66 mg/m^3 comparativement à 1,93 pour la microbalance (TEOM). Quand le taux d'échantillonnage de la microbalance était plus élevé, soit 2,0 litres/min, les résultats ont été de 2,37 mg/m^3 comparativement à 1,26 mg/m^3, respectivement. La vitesse de l'écoulement semble influer sur le rendement de l'appareil. La microbalance (TEOM) possède une haute résolution de masse que l'on a vérifiée en la pesant. Le principe semble logique cependant, et le modèle de production s'avérera peut-être le meilleur moyen pour mesurer la suie en temps réel.

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INTRODUCTION

An air quality investigation related to the performance of catalytic purifiers and ceramic filters was undertaken during late 1985 in Noranda's Brunswick Mine, near Bathurst, New Brunswick. This work is reported in (1,2). For the first time the CANMET monitoring team used two sets of gravimetric samplers placed at the same location (at the top of a raise between two levels judged to be characterized by complete mixing); three of those were fitted with cyclone pre-separators, and three were without.

The result was that the cyclones removed 36% of the combustible material that was airborne, and which the non-cyclone filters collected. Because it was generally acknowledged that diesel soot is 100% respirable and would therefore pass through the cyclones, it was assumed that this non-respirable material was drill oil mist plus oil leakage evaporated from hot machine surfaces, as it was known that drilling activities were in process during the measurements.

Because the engine had been steam-cleaned before these tests were undertaken (i.e. no oil from hot surfaces), and because it is certain that no airborne particulate material other than soot is present in engine exhaust, it was decided to determine the cyclone characteristic under these simplified controlled laboratory conditions which simulate real world dilution of exhaust.

DESCRIPTION OF THE EXPERIMENTAL EQUIPMENT

A schematic drawing of the array of equipment and the arrangement of this equipment during the three test runs is given in Figure 1. Three types of particulate sampling equipment were investigated: 1) gravimetric sampling filters with no inlet devices attached (three samples taken simulatenously), 2) gravimetric sampling filters with standard

cyclones attached to the inlet (likewise, three samples taken simultaneously), and 3) the TEOM Particulate Mass Monitor on loan from the United States Bureau of Mines (three of the six tapered element samplers were made operational). A total of nine samples were, therefore, taken at the same time and in the same location for comparison purposes.

This array of sampling point gear was placed in the centre of the plan cross-section of a vertical duct measuring 0.70 m by 0.59 m (area of cross-section is 0.41 m^2), which conveyed the exhaust air plus engine exhaust from the engine test cell (see photo A of Figure 2). The sampling points or cyclones for the nine samplers were uniformly distributed around a 15.2 cm diameter pitch circle on a holder supported by a centre-line diluted gas sampling probe (see photo B of Figure 2). Each set of three sample points were separated by 120 degrees. The height of the three non-cylone sampling points above the holding plate was made identical to the remaining six cyclone sampling points by the use of a small piece of tygon tube (see photo C of Figure 2) with a similar size opening in order to minimize sampling differences due to apparatus configuration. The array of Dupont sampling pumps, TEOM elements, and sample extraction points from the duct, are shown in Photo A of Figure 3. These pumps were calibrated at 4.0 L/m for the gravimetric samples and at 0.12 L/m for the TEOM for the first day, and 2.0 L/m for the following two days.

The gravimetric sampling cassettes employed 37 mm diameter Gelman A/E type fibreglass filters. The filter weights were measured with electronic scales capable of a resolution of 0.01 mg.

The flow in the vertical duct was a nominal 3.40 m^3/s (7200 scfm) at full fan speed, or 1.70 m^3/s (3600 scfm) at half speed. The lower fan speed was selected yielding a flow somewhat higher than that expected (2.14 m^3/s or 4460 scfm). To establish the exhaust dilution ratio, three methods were employed: 1) measurement of CO in undiluted exhaust and at the vertical duct sampling point in the diluted exhaust, 2) likewise for CO_2, and 3) by calculation employing the undiluted exhaust temperature and the temperature of the mixed air plus exhaust in the vertical duct.

Measures were taken to minimize the leakages in test cell system. The equations used and the data are recorded in Table 1. The undiluted CO analyzer produced meter readings which constantly varied between, for example, 18 to 26 in a short period during one test. It also gave a significantly higher level of dilution then the other two methods and was thought to be less reliable. The average dilution ratio for the CO_2 and flow temperature methods was 24.7:1. The results of these measurements are given in Table 1. This number was confirmed by independent measurements of at a later date at 24.5.

With this exhaust air from the test cell, the exhaust of a Deutz F6L 912W air-cooled, nominal 67 kw (90 bhp) engine, was mixed. The engine was operated at steady-state conditions of 2100 rpm and an output torque of 176 N.m (130 lb.ft), i.e. approximately 60% of full load torque and 90% of full load speed. Under these load conditions the engine exhaust flow was measured to be 370 kg/hr (816 lb/hr) or 0.0854 m^3/s (181 scfm). Therefore, the ventilation air flow was measured to be 9100 kg/hr or 2.10 m^3/s (20,070 lb/hr or 4460 scfm).

The engine is shown on its mount in the test cell in Photo B of Figure 3. Note that the engine exhaust was emitted directly into the incoming ventilation flow near the upper RHS of the photo, in order to facilitate mixing. Photo C of Figure 3 shows the CO_2 and CO diluted gas analyzers and the TEOM computer, keyboard and printout, located in the control room.

Table 1 - Dilution ratio determination

Method	Item		Values			
	test number		1	2	3	Average
CO_2 dilution	duct CO_2	(ppm)	0.280	0.275	0.283	
	exhaust CO_2	(ppm)	6.50	6.30	6.43	
*1	CO_2 dilution ratio		24.6	24.3	24.0	24.3
CO dilution	duct CO	(ppm)	5.5	5.3	5.9	
	exhaust CO	(ppm)	167	147	162	
*2	CO dilution ratio		28.4	25.8	25.7	26.6
exhaust temp dilution	wet gas flow	(lb/hr)	820	812	802	
	exhaust gas temp	(°F)	665	675	680	

Table 1 - (Cont'd)

Method	Item		Values			
	datum air temp	(°F)	70	70	70	
	specific heat	(BTU/lb °F)	0.27	0.27	0.27	
	mixture temp	(°F)	91	95	99	
	specific heat	(BTU/lb °F)	0.25	0.25	0.25	
3	temp dilution ratio		29.6	24.8	20.8	25.1

*1 dilution ratio = $Wa/Wg = \frac{\rho a}{\rho g} \times \frac{CO_2 \text{ gas} - CO_2 \text{ mix}}{CO_2 \text{ mix} - CO_2 \text{ air}}$ (CO_2 air = .035%)

*2 dilution ratio = $Wa/Wg = \frac{\rho a}{\rho g} \times \frac{CO \text{ gas} - CO \text{ mix}}{CO \text{ mix}}$ (CO air = 0)

NOTE: the CO meter reading varied from 18 to 26 meter divisions in a short period on December 18, 1986 (Day #3). CO dilution ratio less reliable.

*3 dilution ratio = $Wa/Wg = \frac{Cg\ (tg - to)}{C \text{ mix}\ (tmix - to)} - 1$

where Wg-exhaust gas flow; Wa-air flow; Wmix=Wg+Wa; ρ is density; C-specific heat and t-temperature.

Table 2 - Gravimetric sampling results (mg)

Test no and cyclone	Calculated non-combustible filter wt (1=3-2)	Measured avge filter combustible (2)	Measured filter tare weight (3)	Calculated particulate deposited (4=5-3)	Measured filter gross (5)	Calculated combustible material (6=5-7)	Measured filter wt combusted (7)	Calculated non-combustible particulate (8=7-1)
1 no cyclone			70.31		72.01		70.18	
			69.68		71.38		69.54	
			72.03		73.44		71.83	
avge	70.55	0.12	70.67	1.61	72.28	1.76	70.52	-0.03
cyclone			71.88		73.62		71.74	
			71.26		73.03		71.19	
			61.26		62.90		61.06	
avge	68.01	0.12	68.13	1.72	69.85	1.85	68.00	-0.01
2 no cyclone			62.14		63.90		62.00	
			61.10		62.89		60.99	
			59.54		61.38		59.52	
avge	60.81	0.12	60.93	1.79	62.72	1.86	60.84	+0.03
cyclone			71.20		73.00		71.04	
			71.53		73.36		71.41	
			69.25		71.06		69.16	
avge	70.54	0.12	70.66	1.81	72.47	1.93	70.54	0
3 no cyclone			63.69		65.29		63.60	
			61.83		63.51		61.69	
			60.51		62.17		60.44	
avge	61.89	0.12	62.01	1.65	63.66	1.75	61.91	+0.02
cyclone			68.60		70.27		68.54	
			65.91		67.58		65.92	
			66.74		68.36		66.63	
avge	66.96	0.12	67.08	1.66	68.74	1.71	67.03	+0.07
AVERAGE SOOT COLLECTED		no cyclone		1.68				
		cyclone		1.73				

DESCRIPTION OF THE EXPERIMENTAL PROCEDURES

The gravimetric filters were placed in pyrex glass containers with covers on and placed in an oven at 150°C (302°F) for a period of 2 hours to drive off excess moisture. They were then removed and weighed using the Perkin-Elmer AD2B micro Autobalance, to a 0.01 mg resolution, and placed in the sampling cassette assemblies. After sampling, they were removed from the cassettes, placed back into the pyrex containers and placed in the oven at 150°C (302°F) for a period of 2 hours. They were removed from the oven, weighed, placed back into the pyrex containers, and then combusted in an electric oven at 500°C (932°F) for 2 hours. After combusting the filters were placed in the oven at 150°C (302°F), left for 2 hours, reweighed and discarded.

TEOM filters were taken from a supply obtained from the Rupprecht and Potashnik Company. These were weighed each morning using the above balance, at room temperature and humidity along with one filter kept as a reference. At the end of the sampling period the TEOM filters were reweighed along with the reference filter. The difference was the collected soot, and the reference filter indicated changes in room temperature and humidity corrections if any. The TEOM equipment was turned on well in advance of the start of each test.

Each day the engine was started and operated at the steady-state conditions described above. The pump sampling flows were calibrated. Particular note was made of the start and stop times of the sampling pumps. All analyzers for the undiluted exhaust and the diluted mixture flow from the test cell, were calibrated. A set of all readings was taken at the start, mid-point and end of each test each day, and averaged values were used when appropriate.

DISCUSSION OF THE RESULTS

The results of the gravimetric sample weighing are given in Table 2. Analysis of Table 2, and reference to Fig. 4 shows that column 4

records the soot accumulated during the sampling period because the calculated non-combustible is essentially zero. Note that the combusted filter weight of column 7 includes some weight loss from the filter itself - 0.12 mg - determined by averaging the weight loss experienced by 4 uncontaminated filters combusted in the oven under the same conditions as the sample filters. This was determined to be 0.29 mg on another occasion, suggesting that this is a variable and care is required to obtain a representative number. Employing the soot figures of column 4 produces concentrations of soot in the vertical duct given in Table 3.

Table 3 - Soot concentrations in the diluted flow

test no	cyclone (yes/no)	start time (h:m)	stop time (h:m)	time diff'l (h:m)	soot weight (mg)	sample flow (L/m)	soot conc't'n (mg/m³)
1	no	8:45	11:28	2:43	1.61	4.0	2.47 *1
	yes				1.72		2.66
2	no	8:25	11:33	3:08	1.79	4.0	2.38
	yes				1.81		2.41
3	no	8:33	11:30	2:57	1.65	4.0	2.33
	yes				1.66		2.34
averages	no				1.68	(-0.07/+0.11)	2.39
	yes				1.73	(-0.07/+0.08)	2.47

*1 average soot concentration = $\frac{1.61 \text{ mg}}{163 \text{ min}} \times \frac{\text{min}}{4.0 \text{ L}} \times \frac{1000 \text{ L}}{\text{m}^3} = 2.47 \text{ mg/m}^3$

The major point that emerges from these results is that the use of a pre-separating cyclone appears to make no impact on the gravimetric sampling values when only diesel soot is involved. The average soot sample weights in Table 3 show that the difference between weights with and without cyclones is 0.05 mg (0.03%), which is less that the within sample variation shown. Therefore, the assumption that diesel soot is 100% respirable appears to be excellent.

This, in turn, suggests that the difference encountered in the field during the Brunswick Mine lead-zinc investigation (2), is due to the presence of other combustible material, presumably drill oil mist or other evaporated oil or fuel from hot surfaces. During the COMINCO lead-zinc investigation (3), the ore was sampled and combusted with no change in weight. It is assumed that the Brunswick ore was likewise inert from the standpoint of a combustion test.

The corresponding concentrations of soot in the diluted air flow varies from 2.39 mg/m^3 for the no cyclone case to 2.47 mg/m^3 for the cyclone case. Back-calculating to determine the undiluted soot concentration in the exhaust gives 25 X 2.39 = 60 mg/m^3 which is in the middle of the known variation of soot concentration for this engine.

The corresponding TEOM results are given in Table 4.

Table 4 - TEOM soot sampling results cyclones employed (mg)

Test no	Measured filter tare weight	Measured filter gross weight	Calculated particulate deposited	TEOM weight output	Sample flow (L/m)	Soot *1 conc't'n (mg/m^3)
1	48.93	48.97	0.04	0.0358		
	46.42	46.46	0.04	0.0348		
	47.69	47.73	0.04	0.0422		
	average			0.0376	0.12	1.93 *2
2	45.74	46.16	0.42	-		
	47.24	47.70	0.46	-		
	48.68	49.11	0.43	-		
	average		0.437		2.00	1.16
3	45.82	46.32	0.50	-		
	49.00	49.27	0.27 (error)	-		
	44.83	45.30	0.47	-		
	average		0.485		2.00	1.37

*1 see sampling intervals in Table 3.

*2 average soot concentration $= \frac{0.0376 \text{ mg}}{162 \text{ min}} \times \frac{\text{min}}{0.12 \text{ L}} \times \frac{1000 \text{ L}}{\text{m}^3} = 1.93 \text{ mg/m}^3$

The TEOM unit provided computer output for only the first test. Vibration was thought to have interferred with the output for the latter two tests. However, the first test results suggest high resolution for the TEOM element weighing system which was roughly checked to the second decimal place by balance weighing as shown. The 0.12 L/m flow was suggested in the manual but such a flow is non-standard in US coal mines and the separator is thought to function properly at the 2.0 L/m flow rate. Thus, increasing the amount collected by increasing the flow to 2.0 L/m as also suggested in the manual yielded higher amounts of soot suitable for the resolution of the balance.

The results suggest that the sampling physics of these circumstances produces flow dependent concentrations for the TEOM, as the latter two tests produce concentrations for the higher flow which are half the gravimetric sample results. It was noticed that the soot collected tended to concentrate near the middle of the TEOM filters with a lesser concentration at the periphery.

RECOMMENDATIONS

(1) If possible, the tests should be rerun at the 2.0 L/m flow when using the gravimetric samplers both with and without cyclones. The use of the higher 4.0 L/m sampling flow would tend to increase the difference between the with and without cyclone results. As there is virtually no difference at the higher flow, it is not likely that this result would change at the lower flow.

(2) Likewise, the TEOM tests should be rerun if possible utilizing the computer only at the beginning and the end of each test in a non-vibrational environment, i.e. during the sampling period, the computer would not be connected to the sampling cassettes.

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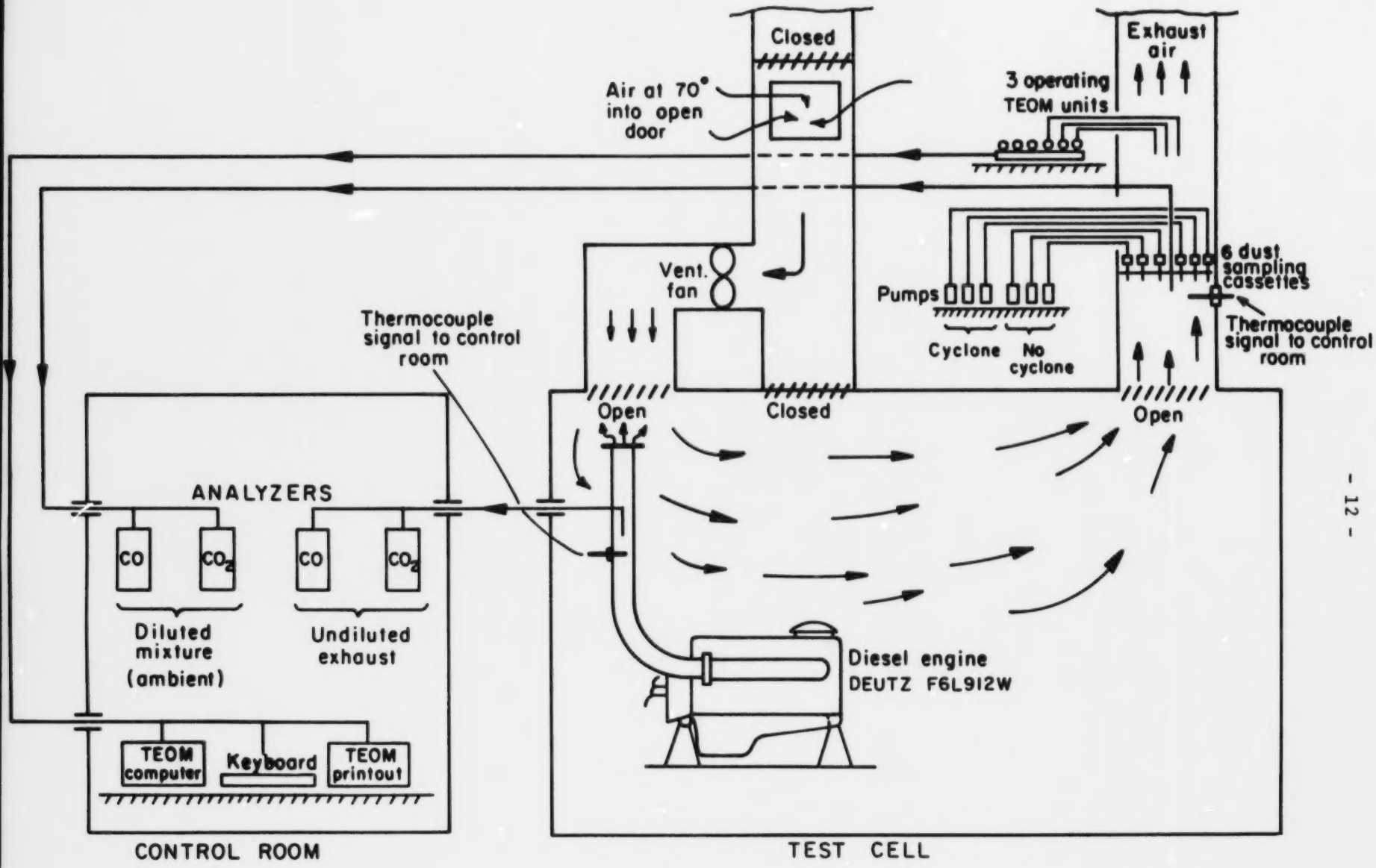

Fig. 1 - Schematic of test equipment arrangement

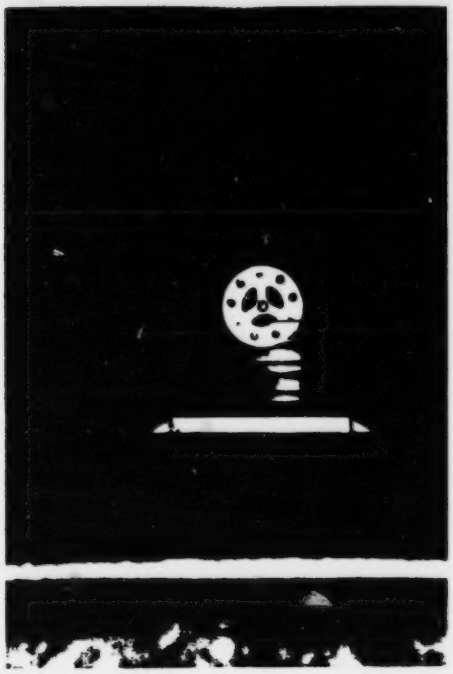

Fig. 2 - Photo A: Orientation of cassettes at sampling point

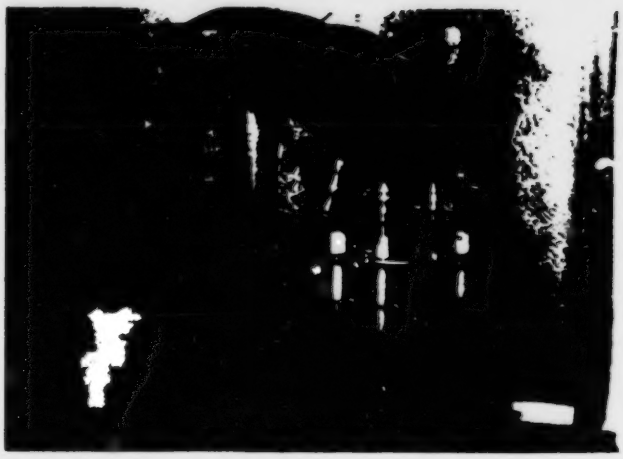

Fig. 2 - Photo B: Closeup of cassette array

Fig. 2 - Photo C: Orientation of soot sampling array relative to duct wall

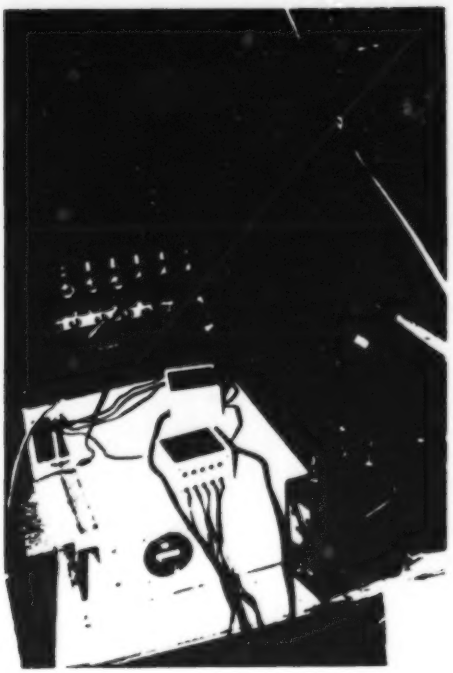

Fig. 3 - Photo A: TEOM, pumps and duct sampling points

Fig. 3 - Photo B: Engine in test cell

Fig. 3 - Photo C: TEOM computer & analyzer array in control room

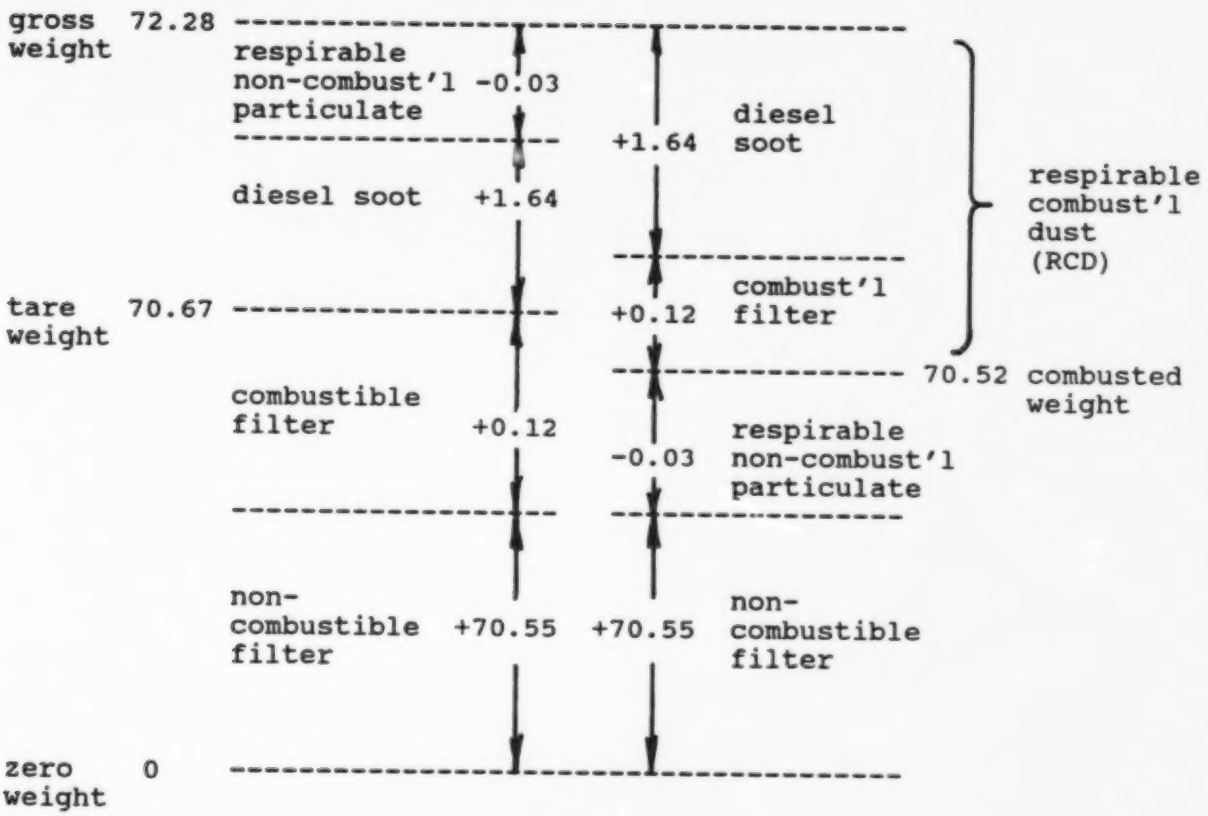

Fig. 4 - Filter/Dust Composition Diagram